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Water Atomization of Barium Fluoride-Calcium Fluoride for Enhanced Flow Characteristics of PS304 Feedstock Powder Blend

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June 2005

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Document Change History

This printing, numbered as NASA/TM—2003-212125/REV1, June 2005, replaces the previous version, NASA/TM—2003-212125, February 2003, in its entirety. It contains the following changes:

Table 2, the numbers have been changed in the last column entitled “Mean Particle Size, μm.”

This report contains preliminary findings, subject to revision as analysis proceeds.

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1. Abstract

PS304 is a plasma spray deposited solid lubricant coating with feedstock composed of NiCr, Cr_2O_3, Ag and BaF_2-CaF_2 powders. The effects of *rounded* BaF_2-CaF_2 particles on the gravity-fed flow characteristics of PS304 feedstock have been investigated. The BaF_2-CaF_2 powder was fabricated by water atomization using four sets of process parameters. Each of these powders was then characterized by microscopy and classified by screening to obtain 45 to 106 μm particles and added incrementally from 0 to 10 wt% to the other constituents of the PS304 feedstock, namely nichrome, chromia and silver powders. The relationship between feedstock flow rate, measured with the Hall flowmeter, and concentration of fluorides was found to be linear in each case. The slopes of the lines were between those of the linear relationships previously reported using *angular* and *spherical* fluorides and were closer to the relationship predicted using the rule of mixtures. The results offer a fluoride fabrication technique potentially more cost-effective than gas atomization processes or traditional comminution processes.

2. Introduction

PS304 is a high temperature composite solid lubricant coating [1–6] developed for the reduction of friction and wear in turbomachinery incorporating foil air bearing technology [7–9]. The constituents of PS304 are nickel-chromium (80Ni-20Cr), chromium oxide (Cr_2O_3), silver and eutectic barium fluoride-calcium fluoride (62BaF_2-38CaF_2, hereafter called *fluorides*), which are each prepared in powder form for plasma spray deposition. To reliably deposit the coating, the powder must flow through the powder feed system in a predictable manner, which is strongly dependent upon on the environmental humidity [10] and the physical properties [11,12] of the coating feedstock powder. Specifically, flow of PS304 feedstock was enhanced with increased particle size and sphericity of the fluorides. Gas atomization produced the best flowing particles due to their high degree of sphericity, but each batch yielded only a small percentage of particles that were suitable for processing [12]. Typical sizes for thermal spray deposited powders range from 5 to 200 μm, where optimal processing characteristics are generally obtained over a preferred range of 20 to 100 μm [13]. However, the mean particle size obtained by gas atomization was 17 μm. Water atomization generally produces higher yields of usable powder without necessarily increasing the percentage of fine particles (*fines*) [14]. Additionally, water atomization tends to be less expensive and more suitable to high volume production when spherical particle shapes are not required since water atomization typically produces rounded particle shapes. The purpose of this investigation

was to optimize the yield size of fluorides obtained by atomization techniques and then study the flow characteristics of plasma spray feedstock powders prepared with these fluorides.

3. Background

a. Particle morphology

Powder particles have many different morphological features which influence flow properties, including shape, topography and surface microstructure, depending on their fabrication technique [15]. Representative particle shapes are shown in Figure 1 including angular, rounded and spherical. The best flow properties are generally obtained with spherical particles. A general quantitative measurement is the *aspect ratio*, the ratio of particle length to width. A more descriptive particle shape quantifier is the *degree of true sphericity* (or, simply, *sphericity*), which was defined by Wadell [16] as the surface area of a sphere with the same volume as a given particle divided by the surface area of the particle. Due to the difficulty of measuring surface areas and volumes of small particles, a practical approximation was later offered:

$$\phi = \frac{d_p}{D_p}$$

where d_p is the projected particle diameter and D_p is the diameter of the smallest circle circumscribing the projected image, shown schematically in Figure 2 [17]. Sphericity will have a maximum value of one for a perfect sphere. Scanning electron microscopy is an effective tool for qualitative analysis of particle morphology. With the use of current commercially available image analysis software, quantitative aspect ratio and sphericity measurements may also be obtained.

b. Processing

The powder constituents used in this study were fabricated by either atomization or comminution (crushing) processes. Atomization produces particles by the rapid solidification of droplets of the molten bulk material where gas or water is used to break up the molten stream [18,19]. Resultant particles from these processes are characteristically spherical (Figure 3a) or rounded (Figure 3b) due to the effect of surface tension on the molten material. An example of the comminution process is shown schematically in Figure 3c. This process takes advantage of the naturally occurring flaws in brittle materials to produce a powder from a bulk material by a combination of compressive and shear forces. The result is an irregular particle with faceted surfaces resulting from brittle fracture (Figure 3c). Approximately 70 wt% of the BaF_2-CaF_2 particles produced by comminution were smaller than the usable size [11,12]. This material had to then be melted again and re-processed, adding significant cost to the powder fabrication process. Therefore, modification of the fluoride particle size and shape was identified as the primary focus of this and other recent investigations [11,12].

c. Materials

Figure 4 shows the constituents of PS304 and their physical characteristics are listed in Table 1. The nichrome particles shown in Figure 4a are 44 to 74 μm in size and have a rounded shape resulting from water atomization. The chromia particles shown in Figure 4b have an angular morphology. This powder was fabricated by sintering the bulk material into large bricks and then comminuting the bricks into a powder. The spherical silver particles (Figure 4c) were fabricated by gas atomization. These three powders are available commercially and their sizes and shapes were not modified for this study.

The fluorides were obtained by combining 68 wt% BaF_2 and 32 wt% CaF_2 in a graphite crucible to melt under vacuum at 1100 °C followed by vacuum cooling. The solidified material was then removed from the crucible and crushed into a powder for subsequent processing by atomization techniques. Previously, BaF_2-CaF_2 was fabricated by gas atomization [12], which produced spherical particles. However, the mean yield particle size was approximately 17 μm, while the desired size range was 45 to 106 μm. To the best of the authors' knowledge, BaF_2-CaF_2 has not previously been fabricated by water atomization. This study was undertaken to determine if water atomization could be used to fabricate fluorides and to optimize the yield of these particles for the desired size range (45 to 106 μm).

The principles of a water atomization process are illustrated schematically in Figure 5 [14,18]. In this process, the material is melted in a crucible with a bottom-tapped nozzle under an argon cover. The molten material is allowed to flow under gravity through the nozzle and free-fall for a distance *F*. The atomization fluid then breaks the molten stream up into droplets, which form particles as they quench and solidify. Compared to spherical particles typically produced by gas atomization, water atomization tends to produce merely rounded particles due to the higher thermal conductivity of the atomization fluid [15,19]. The advantages of water atomization over gas atomization are reduced cost, increased production volume and increased mean particle size [14].

4. Experimental Procedure

a. Materials

Materials used in this study were purchased from commercial vendors. Nichrome powder (98.17 percent by chemical analysis) was fabricated by water atomization and screened to retain 44 to 74 μm particles. Chromia powder (99.18 percent by chemical analysis) was fabricated by comminution and screened to retain 30 to 44 μm particles. Silver powder (100 percent purity) of 45 to 100 μm diameter was fabricated by gas atomization. High purity (100 percent) calcium fluoride and barium fluoride (99 percent purity) were combined at the eutectic composition for the binary compound (68 wt%BaF_2-32 wt%CaF_2) in a graphite crucible, melted under a moderate vacuum at 1100°C and then cooled under a moderate vacuum.

Large blocks of the solidified fluoride material (5 to 10cm) were removed from the crucible and comminuted into a coarse powder for subsequent processing by water atomization. Approximately 1060g of the fluorides were loaded into a graphite crucible and induction melted at 1250 °C for processing with four sets of parameters (Processes

A, B, C and D), as listed in Table 2. The four processes differ by the water pressures and water and melt flow rates used.

ASTM standard specification B 214–99 was used to classify the fluorides by size. The screens used in this procedure were manufactured according to ASTM standard specification E–11. The screens were stacked vertically in order of coarsest mesh to finest mesh. The screen mesh sizes used were numbers 140, 170, 200, 230, 270, 325 and 400. The screening instrument uses a vertically oscillating column of air and a combination of vertical and horizontal tappers to separate the particles according to size. The fluorides were classified by screening and a 45 to 106 μm particle size distribution was retained.

Morphology of the fluorides was measured using commercially available image analysis software with images imported from an SEM. A small amount of powder was poured onto waxed paper and tapped gently to allow the particles to come to rest at their most stable position, which will also produce the largest projected area [17]. The particles were distributed so that they were not in contact and then transferred to an SEM mount with double-sided conductive tape. The particle image was distinct from the black background. The software measurement system was calibrated using a micron marker such that each pixel corresponded to 0.56 μm. Each imported monochromatic SEM image had a bit depth of 8-bits per pixel with an image resolution of 712 by 484 pixels [20]. The supplied image analysis software was used to measure the aspect ratio, the two-dimensional surface area of selected particle images, from which d_p was calculated, and the maximum chord length D_p (see Figure 2).

b. Flow test

The flow rate of each powder blend was tested according to ASTM B 213–97. For this test, a 50 g sample of the powder blend being tested was loaded into a Hall flowmeter. For each flow test, a powder blend consisting of 60 g nichrome, 20 g chromia, 10 g silver and from 0 to 10 wt% fluorides was prepared by mixing the constituents together in a 125 mL high density polyethylene bottle until well blended. A 50.0g sample was obtained from the powder blend for flow testing. A digital stopwatch was used to measure the time it took the entire sample to exit the funnel. The test was repeated once; the times were averaged to the nearest 0.1s and designated the *flow time*. The data were reported on a plot of flow time versus the weight fraction of fluorides in the powder blend.

c. Processing

PS304 coating was applied by plasma spray [21,22] to 25.4 mm diameter, 6.35 mm thickness Inconel 718 substrates using standard process parameters [23]. The substrates were prepared by grit blasting with coarse Al_2O_3 particles and then applying the coating at a thickness of approximately 0.4 mm.

5. Results and Discussion

a. Materials

The morphologies of the water atomized fluorides are shown in Figure 6. The particles have the rounded shape characteristic of water atomized materials. The average aspect ratio was 1.62 and the average sphericity was 0.78, based on a 185 particle sample, which is similar to values previously reported for angular fluorides (Table 1) [24]. However, as can be seen by comparing the powder images, sphericity does not necessarily account for differences in surface area. Therefore, particles with higher surface area due to more attached satellites may have the same sphericity as those with lower surface area. At high magnification, a fine grained microstructure may be observed, attributed to rapid solidification. The fluorides have increasingly smoother surfaces due to the decreasing occurrence of attached satellites from Process A to Process D. Apparently, the higher water pressure caused increased breakup of the molten fluorides where smaller droplets impacted larger ones before the larger particles solidified [25,19].

Mean particle sizes as measured by a light blockage particle analysis technique are listed in Table 2 [26]. Compared to the mean particle diameter of 17 μm from previously studied spherical fluorides, the mean sizes of the rounded fluorides are substantially larger, ranging from 31.7 to 46.9 μm.

b. Flow test

The results from flow testing are shown graphically in Figure 7. The flow time increased linearly with increasing concentration of the fluorides. Flow was slightly improved with increasing surface smoothness of the fluorides, as demonstrated by the fact that the fluorides produced by Process A (with more attached satellites and, correspondingly, higher surface area) resulted in generally higher flow times than for Process B fluorides and so on. Therefore, the sphericity measurement may not carry enough relevant information to uniquely indicate differences in flow behavior with the given experimental procedures. Figure 7 also shows lines representing the flow behavior of similarly prepared powder blends with angular and spherical fluorides [12]. The flow times of the powder blends with rounded fluorides are generally between those with angular and spherical fluorides.

Regardless of the possible particle-particle interactions, simply based on the rule of mixtures, the volume of a standard 50 g powder sample of PS304 feedstock powder is expected to increase with increasing content of fluorides because the theoretical density of the fluorides, 4.01 g/cm^3, is less than the other constituents. The densities of nichrome, chromia and silver are 8.57, 5.22 and 10.49 g/cm^3, respectively (Table 1). If the flow time of the powder blend were only dependent upon the volume of powder transferred though the funnel with no particle interaction effects, the flow time of a 50 g sample with 10 wt% fluorides would be expected to take 9 percent longer than a 50 g sample with no fluorides, due to the 9 percent increased volume for the 50 g sample. The calculated flow times based only on increased volume are represented by the dashed line in Figure 7. The powder blend with 10 wt% angular fluorides would then have a flow time of about 29s as

shown by the calculated line in Figure 7, instead of the measured 31.6s, if volume alone were considered. Based on this, the measured flow time using angular fluorides was approximately 9 percent higher than expected. This difference is thought to be due to the increased interparticle cohesion generated by the angular fluorides and its resulting increase on the volume of the powder blend. Using spherical fluorides, the flow time was independent of the concentration of fluorides. In this case, the effect of increasing volume was offset by the improvement in flow created by the spherical fluorides. With rounded fluorides, however, the flow behavior essentially follows the rule of mixtures calculation.

c. Performance

The as-deposited pull-off adhesion strength of the coatings on the Inconel substrates was measured using a standard test procedure [27]. The average adhesion strengths obtained using conventional angular fluorides and rounded fluorides fabricated by process D were 20.2±5.2MPa [27] and 18.7±3.2MPa, respectively. By statistical inference, no difference in the mean values from these two sample populations could be detected. Aluminum witness coupons were coated along with the Inconel substrates to perform analysis of the coating chemistry by an x-ray fluorescence technique. Each coating had nominally the same composition. Coated Inconel samples were sectioned perpendicular to the coating and prepared with standard metallographic procedures to examine the as-sprayed microstructures, shown in Figure 8. The coatings had characteristic lamellar thermal sprayed microstructure with no major differences between them. Tribological performance of the coatings may also be investigated with pin-on-disk testing or bearing friction and wear evaluation [1,4].

6. Conclusions

The objective of this study was to investigate the effects of the addition of rounded fluorides on the flow characteristics of PS304 feedstock. Based on the results, the following conclusions were drawn.

- Water atomization can be used to fabricate novel rounded fluorides, which tend to have aspect ratio and sphericity measurements similar to those of angular fluorides and larger mean diameters than previously studied gas atomized spherical fluorides.
- Further work is needed to develop a metric that quantifies particle morphology in a way that is distinct to flow behavior.
- Increasing water pressure during atomization tends to produce particles with more attached satellites.
- Increasing the concentration of fluorides in the powder blend produces a linear increase in powder flow times.
- Fluorides with smoother surfaces generate less of an increase in the powder flow time.
- The flow behavior of PS304 with rounded fluorides is intermediate to that with angular and spherical fluorides and is approximated by a rule of mixtures calculation.

- The PS304 coating characteristics obtained with rounded fluorides are equivalent to those with angular and spherical fluorides.
- Water atomization is potentially a good method for high volume, low cost production BaF_2-CaF_2 powder with predictable flow characteristics.

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Table 1. Summary of PS304 constituent properties [24].

	Fluorides ($68BaF_2$-$32CaF_2$)			**Nichrome (80Ni-20Cr)**	**Chromia (Cr_2O_3)**	**Silver (Ag)**
Fabrication Method	**Angular**	**Rounded**	**Spherical**			
Surface Morphology	Comminution	Water atomization	Gas atomization	Water atomization	Comminution	Gas atomization
Particle Size Distribution	Angular (AR=1.61, ϕ=0.77)	Rounded (AR=1.62, ϕ=0.78)	Spherical (AR=1.13, ϕ=0.94)	Rounded	Angular	Spherical
Theoretical Density (g/cm^3)	45–106 μm	45–106 μm	45–106 μm	44–74 μm	30–44 μm	45–100 μm

Table 2. Process parameters used to fabricate rounded fluorides.

Process	Water pressure, MPa	Water flow rate, Lpm	Melt flow rate, g/sec	Water-melt ratio	Mean Particle Size, μm
A	19.6	16	28.2	8.9	38.4
B	19.6	16	10.6	23.8	28.8
C	9.17	17	4.2	63.8	61.0
D	9.17	17	8.9	30.1	68.9

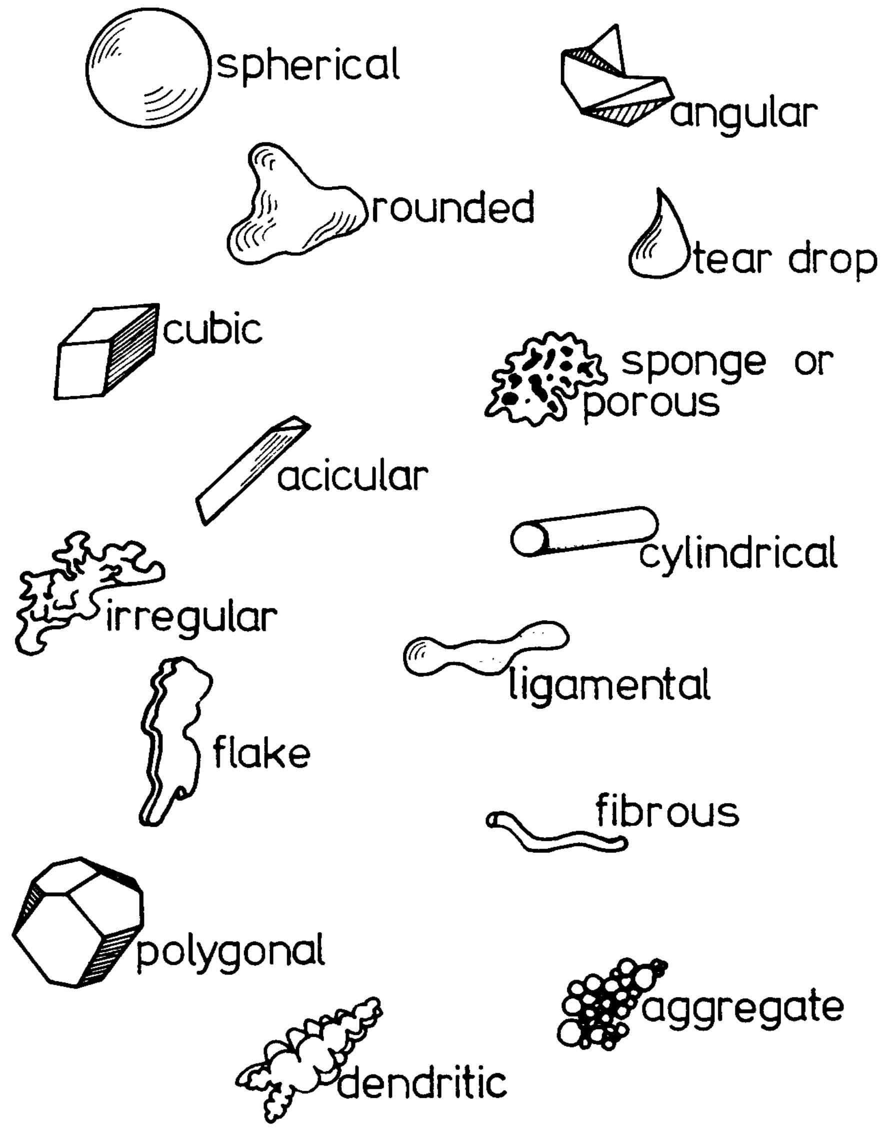


Figure 1. Typical particle shapes [15].

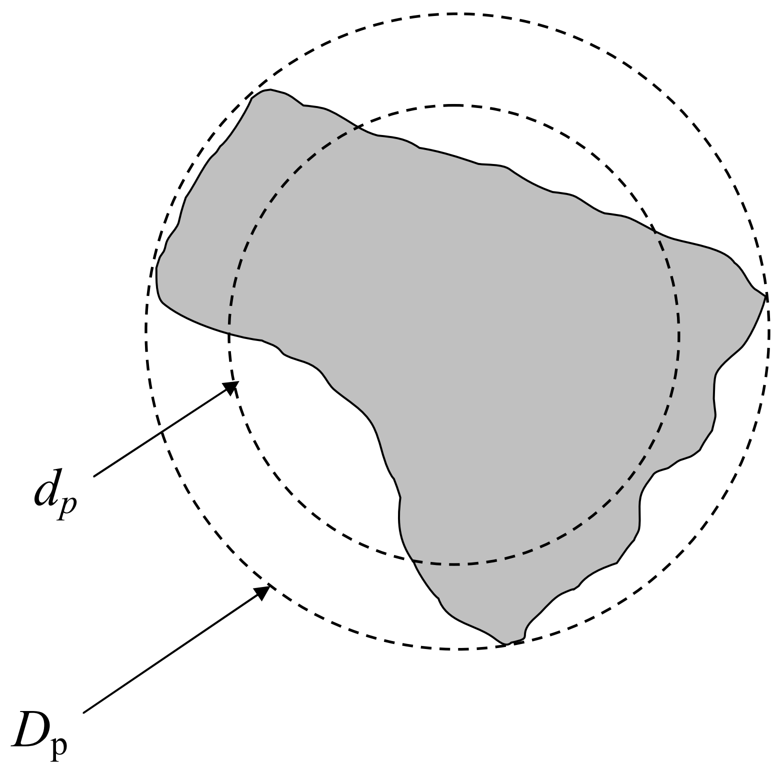


Figure 2. Parameters used in the calculation of sphericity $\phi = \frac{d_p}{D_p}$.

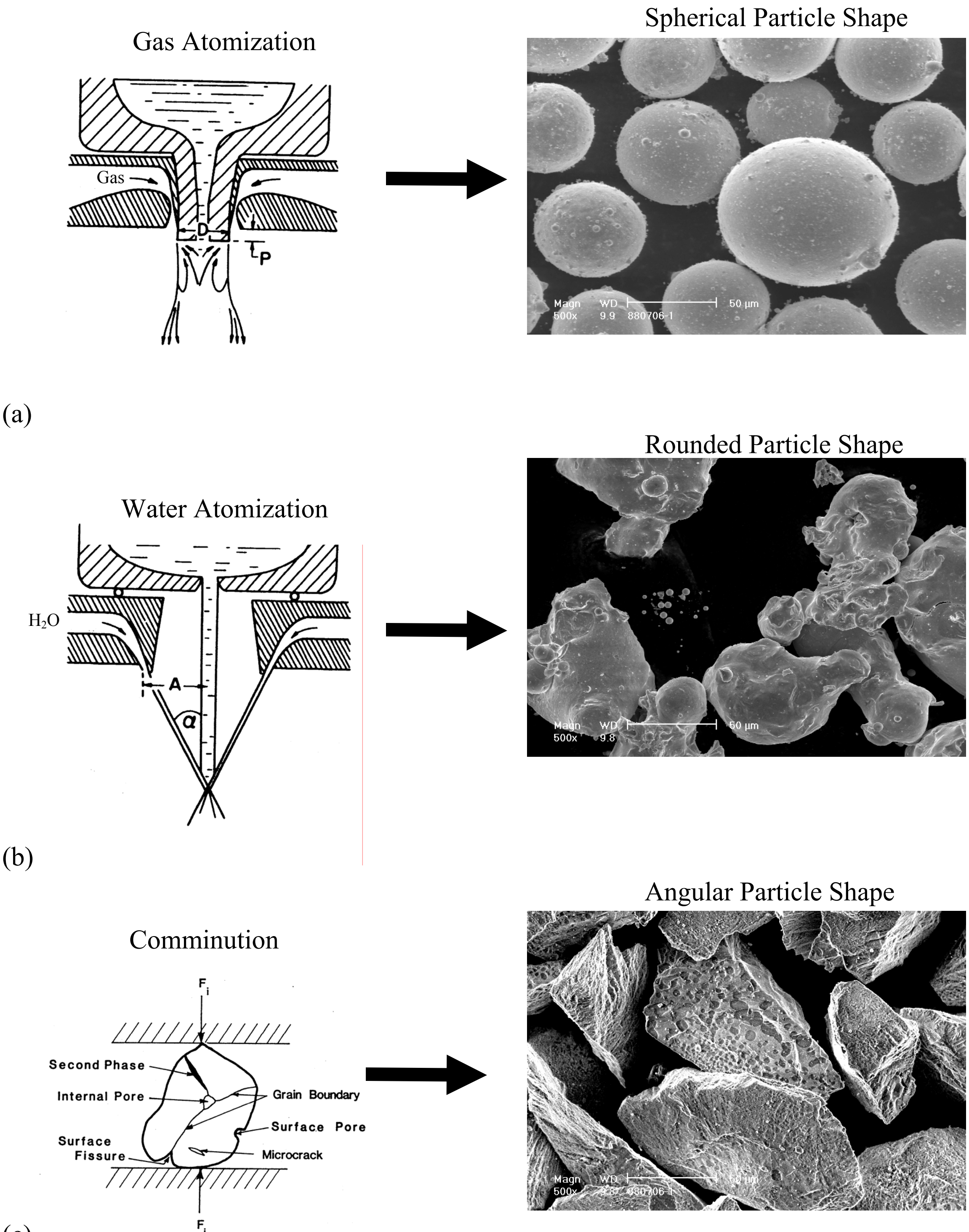
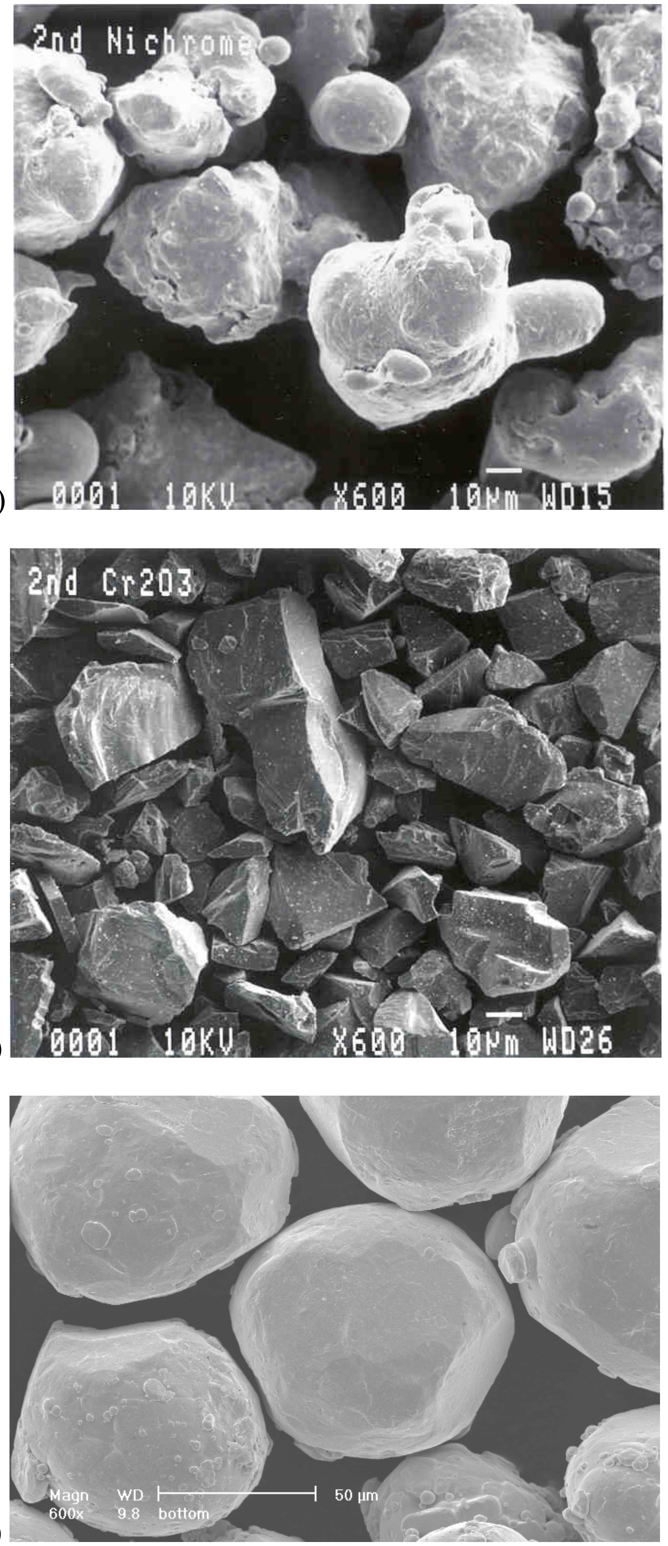


Figure 3. Comparison of particle fabrication techniques considered in this investigation and their resultant powders (original SEM magnification 500×) [28,29].



(a)

(b)

(c)

Figure 4. Non-fluoride constituents of PS304 feedstock; (a) nichrome, (b) chromia and (c) silver (original magnification 600×).

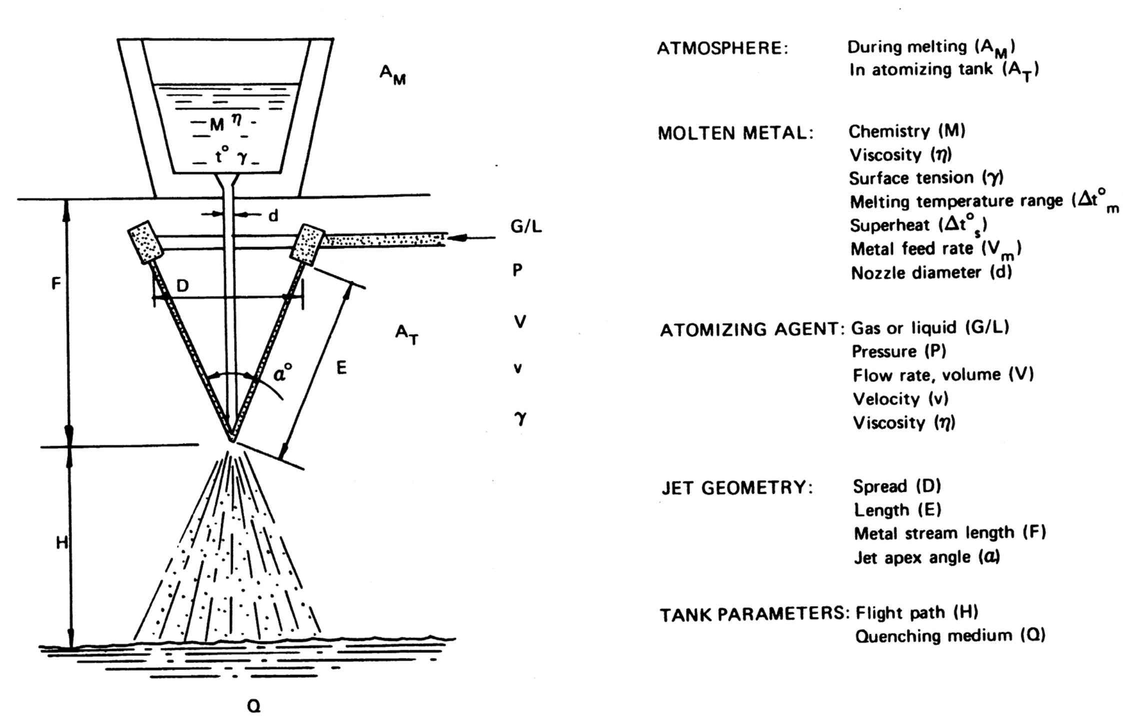
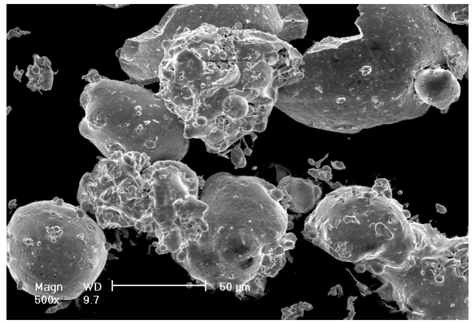


Figure 5. Principle elements of a water atomization process [14,18].

(a)



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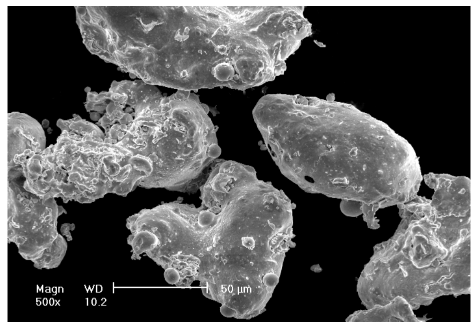

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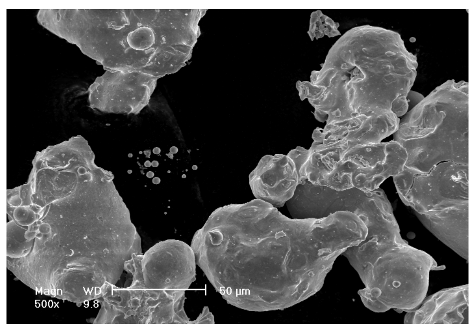

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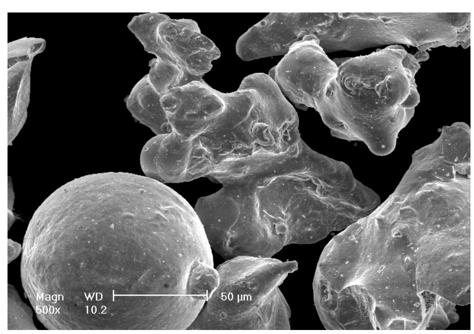


Figure 6. Typical morphologies of fluorides fabricated by (a) Process A, (b) Process B, (c) Process C and (d) Process D from Table 2 (original magnification 500×).

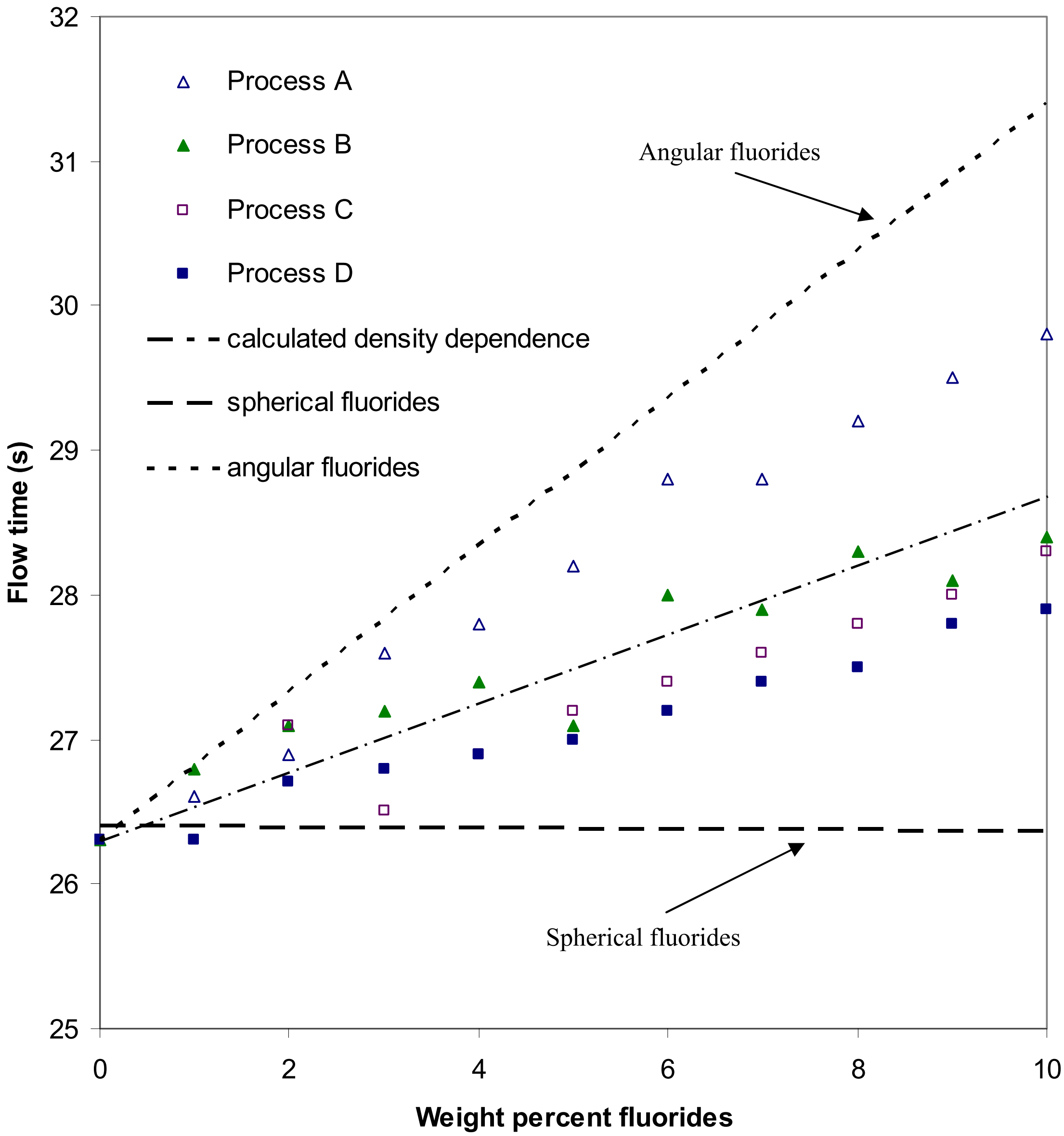
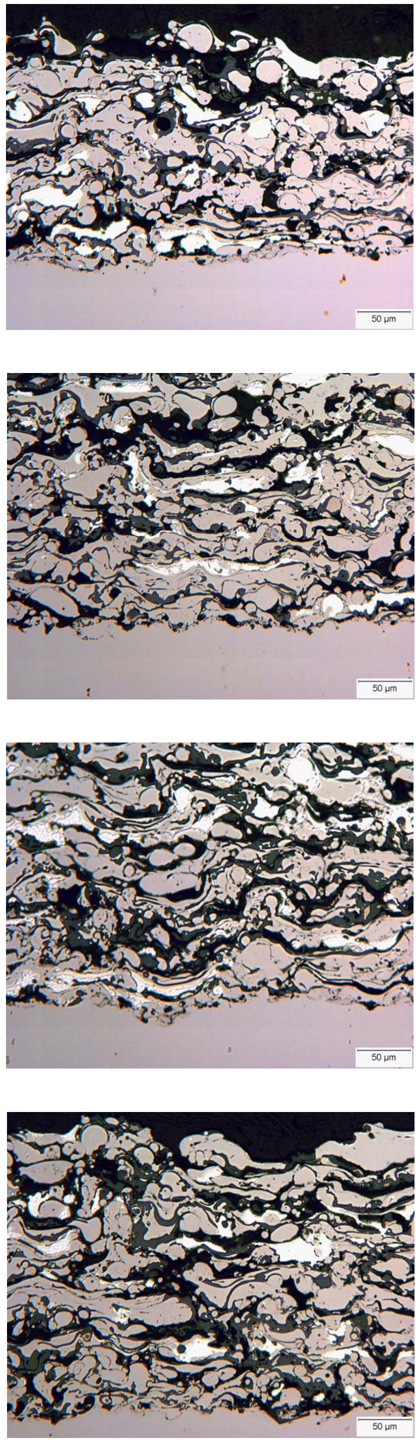


Figure 7. Plot of flow time as a function of BaF_2-CaF_2 concentration. Angular and spherical fluoride concentration dependence from previous work [12].



(a)
(b)
(c)
(d)

Figure 8. Characteristic PS304 coating microstructures with rounded fluorides from (a) Process A, (b) Process B, (c) Process C and (d) Process D (original magnification 200×).

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Water Atomization of Barium Fluoride-Calcium Fluoride for Enhanced Flow Characteristics of PS304 Feedstock Powder Blend

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13. ABSTRACT ***(Maximum 200 words)***

PS304 is a plasma spray deposited solid lubricant coating with feedstock composed of NiCr, Cr_2O_3, Ag, and BaF_2-CaF_2 powders. The effects of rounded BaF_2-CaF_2 particles on the gravity-fed flow characteristics of PS304 feedstock have been investigated. The BaF_2-CaF_2 powder was fabricated by water atomization using four sets of process parameters. Each of these powders was then characterized by microscopy and classified by screening to obtain 45 to 106 μm particles and added incrementally from 0 to 10 wt% to the other constituents of the PS304 feedstock, namely nichrome, chromia, and silver powders. The relationship between feedstock flow rate, measured with the Hall flowmeter, and concentration of fluorides was found to be linear in each case. The slopes of the lines were between those of the linear relationships previously reported using angular and spherical fluorides and were closer to the relationship predicted using the rule of mixtures. The results offer a fluoride fabrication technique potentially more cost-effective than gas atomization processes or traditional comminution processes.

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